

Technical Report ARWSB-TR-17018

High Thermal Conductivity Carbon Nanomaterials for Improved Thermal Management in Armament Composites

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March 2017



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REPORT DOCUMENTATION PAGE				Form Approved OMB No. 0704-0188	
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1. REPORT DATE (DD-MM-YYYY) March 2017		2. REPORT TYPE Technical		3. DATES COVERED (From - To)	
4. TITLE AND SUBTITLE High Thermal Conductivity Carbon Nanomaterials for Improved Thermal Management in Armament Composites				5a. CONTRACT NUMBER	
				5b. GRANT NUMBER	
				5c. PROGRAM ELEMENT NUMBER	
6. AUTHOR(S) Stephen Bartolucci				5d. PROJECT NUMBER	
				5e. TASK NUMBER	
				5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) U.S. Army ARDEC Benet Laboratories, RDAR-WSB Watervliet, NY 12189-4000				8. PERFORMING ORGANIZATION REPORT NUMBER ARWSB-TR-17018	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)				10. SPONSOR/MONITOR'S ACRONYM(S)	
				11. SPONSORING/MONITORING AGENCY REPORT NUMBER	
12. DISTRIBUTION AVAILABILITY STATEMENT Approved for public release; distribution is unlimited.					
13. SUPPLEMENTARY NOTES					
14. ABSTRACT Carbon nanomaterials such as carbon nanotubes, nanofibers, and graphene have shown tremendous potential as additives for polymer matrices. In addition to improving mechanical and electrical properties, these forms of carbon typically demonstrate high intrinsic thermal conductivities, a property that could be useful in improving the thermal dissipation performance of polymer matrix composites. In this study, carbon nanotubes, carbon nanofibers and graphene have been added to polymers and polymer matrix composites in order to study the effect on the thermal conductivity of the composite. It is demonstrated that these species can improve the thermal conductivity. The improved properties are used as inputs to thermal models of a generic composite tubular structure relevant to armament systems in order to study the effect on thermal management during heating cycles.					
15. SUBJECT TERMS Carbon Nanomaterials; Carbon Nanotubes; Nanofibers; Graphene; Thermal Conductivity; Armament Composites					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT U	18. NUMBER OF PAGES 15	19a. NAME OF RESPONSIBLE PERSON Steve Bartolucci
a. REPORT U/U	b. ABSTRACT U	c. THIS PAGE U			19b. TELEPHONE NUMBER (Include area code) (518) 266-5189

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Abstract

Carbon nanomaterials such as carbon nanotubes, nanofibers, and graphene have shown tremendous potential as additives for polymer matrices. In addition to improving mechanical and electrical properties, these forms of carbon typically demonstrate high intrinsic thermal conductivities, a property that could be useful in improving the thermal dissipation performance of polymer matrix composites. In this study, carbon nanotubes, carbon nanofibers and graphene have been added to polymers and polymer matrix composites in order to study the effect on the thermal conductivity of the composite. It is demonstrated that these species can improve the thermal conductivity. The improved properties are used as inputs to thermal models of a generic composite tubular structure relevant to armament systems in order to study the effect on thermal management during heating cycles.

Table of Contents

Abstract	i
Table of Contents	ii
List of Figures	iii
List of Tables	iii
1. Introduction.....	1
2. Experimental Procedure.....	2
2.1 Composite Preparation.....	2
2.2 Thermal Testing	3
2.3 Thermal Modeling	3
3. Results and Discussion	3
3.1 Composite Microstructures	4
3.2 Thermal Conductivity	6
3.3 Thermal Modeling	9
4. Conclusions.....	11
5. Acknowledgements.....	11
6. References.....	12

List of Figures

Figure 1. Scanning electron microscopy images of the fracture surface of the various epoxy-based composites	4
Figure 2: Fracture surface of the Zyvex nanotube matrix material	5
Figure 3: Plot of thermal conductivity for epoxy-based composites with carbon nanofibers (CNF) and graphene platelets (GPL)	7
Figure 4: Thermal conductivities of various composite materials based on the Zyvex resin and fiber system, as well as the addition of N12 nanostitch interlaminar carbon nanotubes	9
Figure 5: Thermal modeling results for a generic thin-walled metallic tube overwrapped with polymer matrix composite	10

List of Tables

Table 1. Epoxy-Based Sample.....	6
Table 2: Zyvex Resin-Based Samples	8

1. Introduction

The Army has spent considerable resources in researching and implementing lightweight composite materials in armament components in order to reduce the weight of systems, decrease logistical burdens, and increase performance. Polymer matrix composites (PMCs) offer low density and high specific strength and stiffness. One disadvantage of PMCs for certain applications is the low thermal conductivity (k) afforded by these materials. In some applications, it is advantageous to dissipate heat from a heat source, in order to keep localized temperatures in the structure lower, and more evenly distributed. While PMCs generally have low thermal conductivities, there are some methods that can be used to increase the heat dissipation capabilities of these materials.

The addition of high k conductive species to a polymer matrix can often result in increased overall thermal conductivity of the composite. Some high k materials, such as metals, can be made into powder form and mixed into the polymer. The main disadvantages of this approach are that the polymer and metal usually form a weak interface and the high-density metals add considerable weight to the composite, since filler levels are typically around 30 vol%. Recently, the development and commercialization of carbon-based materials such as carbon nanotubes [1] and graphene [2], has led to the development of nano-carbon PMCs. Carbon nanotubes (CNTs) [3,4] and specifically, individual sheets of graphene, have the highest theoretical k values of any known material (5300 W/m-K) [5], an order of magnitude higher than copper (400 W/m-K) and four orders of magnitude higher than typical polymers (0.1 - 0.5 W/m-K). These low-density materials have the capability of greatly increasing the k of the composite, while adding no significant weight to a structure. Recent research on this topic has had mixed results, with improvements in k for the nanocomposite typically ranging from 10% to 100%. [6] Some have achieved over an order of magnitude increase in k of the composite compared to the pure polymer. Aligning the high aspect ratio carbon nanotubes can increase the thermal conduction in the direction of alignment. Alignment has been achieved in PMCs under high magnetic or electric fields during curing of the polymer. [7-9]. Composites with graphene core-shell structures [10] and graphene paper [11] have shown great promise for improved thermal conductivity.

In this report, the performance of nanotube, nanofiber, and graphene additives to polymers and polymer matrix composites is presented.

2. Experimental Procedure

2.1 Composite Preparation

All composite specimens were prepared in the laboratory at Benet. Epoxy-based composites were made using Epoxy 2120 (Fibre Glast) resin and hardener system. Carbon Nanofibers (Pyrograf Products, PR-24-HHT-LD) were added to the epoxy at loadings of 0, 0.5, 1.0 and 10.0 wt%. Carbon nanofibers (CNF) were dispersed in acetone for 90 minutes using bath sonication followed by 90 minutes of sonication with the resin. The acetone was removed from the CNF/resin mixture by heating on a hot plate at 70 °C under stirring for 3 hours and then drying under vacuum for 20 hours at 80 °C. The hardener was then added to the CNF/resin mixture using a high-speed mixer (Flacktek) at 2000 rpm for 1 minute and then at 3500 rpm for 1 minute. The material was degassed in vacuum for 30 minutes and then poured into a silicone and cured overnight under 40 psi pressure to force out any bubbles trapped during pouring into the mold. The samples were post-cured at 80 °C for 2 hours. Samples were cut and sanded to the appropriate dimensions for thermal conductivity testing. The 1.0 wt% graphene nanoplatelet (GPL) composite was prepared in the same manner as the CNF composites. Fracture surfaces of samples were imaged using an FEI Nanolab 600i Dual Beam Field Emission Scanning Electron Microscope operating at 10kV.

Carbon fiber (37-800, 30K)/epoxy composites were fabricated with a specialty resin by Zyvex Corporation (Arovex SC prepreg system) using traditional prepreg composite fabrication techniques. This resin matrix contains non-covalently functionalized multi-walled carbon nanotubes (0.4 wt%). In addition, composites using the Zyvex resin were also fabricated with the addition of nanostitch vertically-aligned carbon nanotube forest interlaminar layers (N12 Technologies) between the Zyvex prepreg layers in order to study the effect on thermal conductivity. Samples were made of the (1) neat Zyvex epoxy resin, (2) Zyvex resin with the nanotubes included, (3) Zyvex resin with 37-800 carbon fibers, (4) Zyvex resin (with nanotubes) and 37-800 carbon fibers, (5) Zyvex resin (with nanotubes) and 37-800 fibers cured under autoclave, (6) Zyvex resin with 37-800 carbon fibers with N12 nanostitch layers in the interlaminar region, (7) Zyvex resin (with nanotubes) and 37-800 carbon fiber with N12 nanostitch layers in the interlaminar region. Samples (1) and (2) were cured in an oven at 121°C using Omicure U-24M and Omicure DDA-10 curing agents. Samples (3), (4), (6) and (7) were cured in an oven at 121°C and sample (5) was cured in an autoclave at 121°C under pressure at 100 psi. Curing in the autoclave results in a more compact structure. By fabricating these different types of samples, we can elucidate the contribution of each additive (resin, nanotubes, carbon fibers, nanostitch layers) to the improvement in thermal conductivity.

2.2 Thermal Testing

Thermal diffusivity, α , was measured using a laser flash technique at the Thermophysical Properties Research Laboratory (TPRL) (West Lafayette, IN) and at Netzsch Instruments Applications Laboratory (Burlington, MA) using a NETZSCH LFA 467 Hyperflash instrument. Bulk density (d) values were calculated from the sample's geometry and mass. Specific heat (C_p) was measured using differential scanning calorimeters (at TPRL) and the LFA 467 Hyperflash instrument at Netzsch. Thermal conductivity k values were calculated as a product of these quantities, i.e. $k = \alpha \times C_p \times d$. Thermal diffusivity, α , is determined using the laser flash diffusivity method conforming to (ASTM E1461-13). The density values are estimated to be accurate to within 1%, the thermal diffusivity values within 5%, the specific heat to within 5% and the calculated thermal conductivity values to within 7% (+/-). All Zyvex samples were tested at Netzsch at 50 °C and all epoxy samples were tested at TPRL at 80 °C.

2.3 Thermal Modeling

In order to show the effect of improved thermal conductivity of a composite on a geometric and material structure relevant to armament systems, an ABAQUS model of a generic thin-walled metallic tube with a polymer matrix composite overwrap is presented. The model was examined using a typical thermal conductivity of a PMC and then with a thermal conductivity that is 100% higher.

3. Results and Discussion

In this work, we examine different scenarios for the addition of nanocarbon species to polymeric materials. In the first scenario, we add low-cost nanocarbon materials to epoxy thermoset polymer; CNF and GPL were mixed into the epoxy. In the second scenario, we add carbon nanotubes to a thermoset/carbon fiber composite to test how the addition of the nanotubes affects the thermal conductivity. In some applications, a pure polymer is sufficient, where simple additions of nanocarbon species will result in thermal property improvements and, perhaps, mechanical properties as well. In other cases, a simple polymer matrix will not have the mechanical properties that are needed for the desired application. For this case, carbon fibers are necessary to give the desired strength and stiffness. We have added carbon nanotubes to these composites in the both the matrix and the interlaminar areas in order to improve thermal conductivity.

3.1 Composite Microstructures

The microstructures for the samples in the first scenario are shown in Figure 1. The 0.5 and 1.0 wt% CNF fracture surfaces show some areas of CNF agglomeration (lighter colored areas approximately 20 microns in size). There are still single CNFs dispersed throughout the matrix as seen in Fig. 1c and 1e. The 10 wt% CNF sample in Fig. 1f and 1g show a surface that is nearly completely covered in nanofibers, with only minor agglomeration. The 1 wt% GPL surfaces in Fig. 1h and 1i show regions of GPL covering the surface (wrinkled morphology).

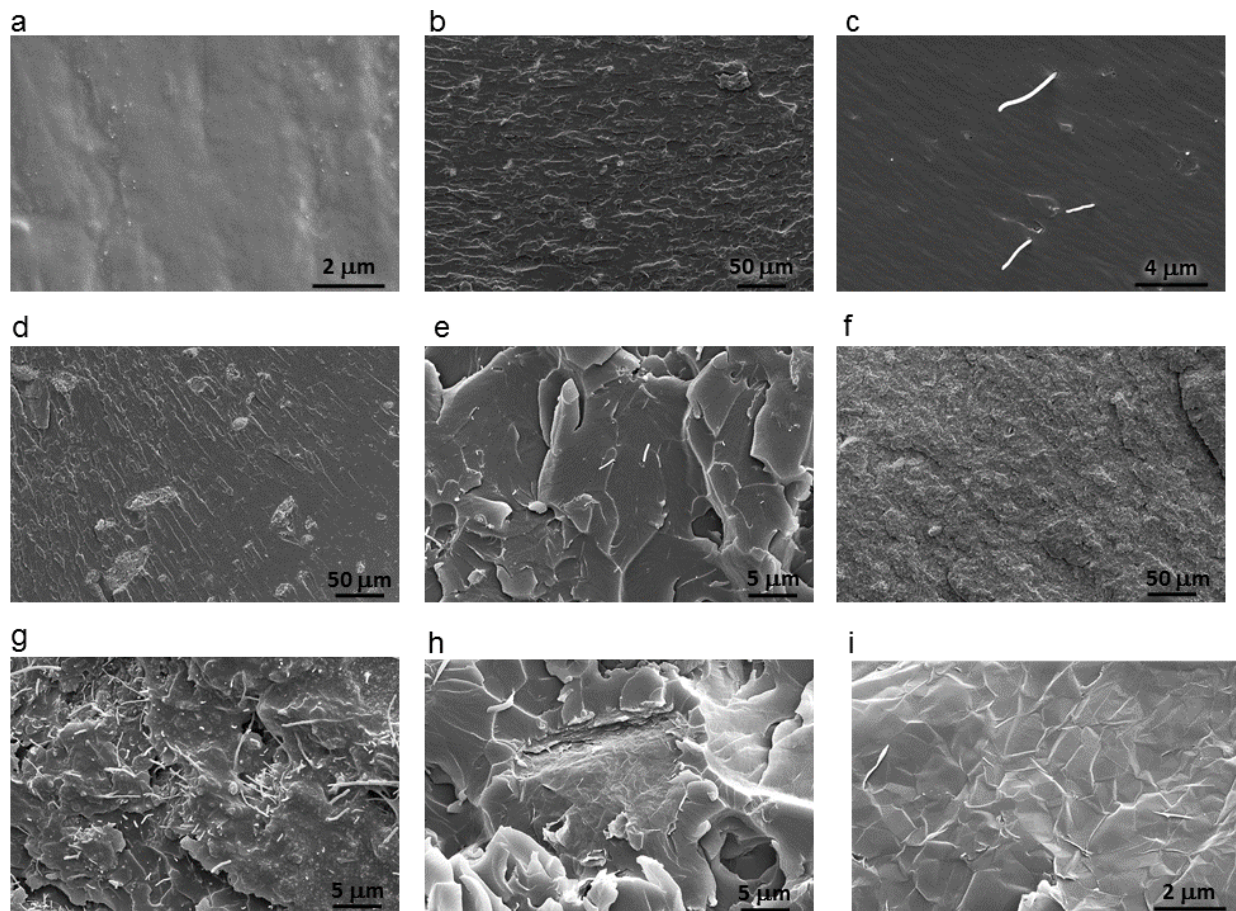


Figure 1. Scanning electron microscopy images of the fracture surface of the various epoxy-based composites. a) pure epoxy, b) 0.5 wt% CNF with lighter areas being agglomerates of CNF c) 1 wt% CNF surface showing several nanofibers protruding d) 1.0 wt% CNF fracture surface with lighter areas being agglomerates of CNF e) 1 wt% CNF surface showing individual CNF protruding from surface f) 10 wt% CNF surface, showing CNF covering large portion of the fracture surface g) 10 wt% CNF surface showing many CNF protruding from surface h) 1 wt% GPL surface showing GPL on surface i) 1 wt% GPL surface covered by GPL (higher magnification of area in h).

Figure 2 shows the Zyvex with CNT matrix material (0.4 wt% CNT). Multi-walled nanotubes are seen protruding from the fracture surface. There are some visible agglomerates of CNTs on the surface. Fig 2d shows the hybrid composite fracture surface with carbon fibers, where the fiber direction is perpendicular to the page in region 1, and parallel to the page in region 3. Region 2 denotes where the N12 carbon nanotube layer is in the interlaminar region.

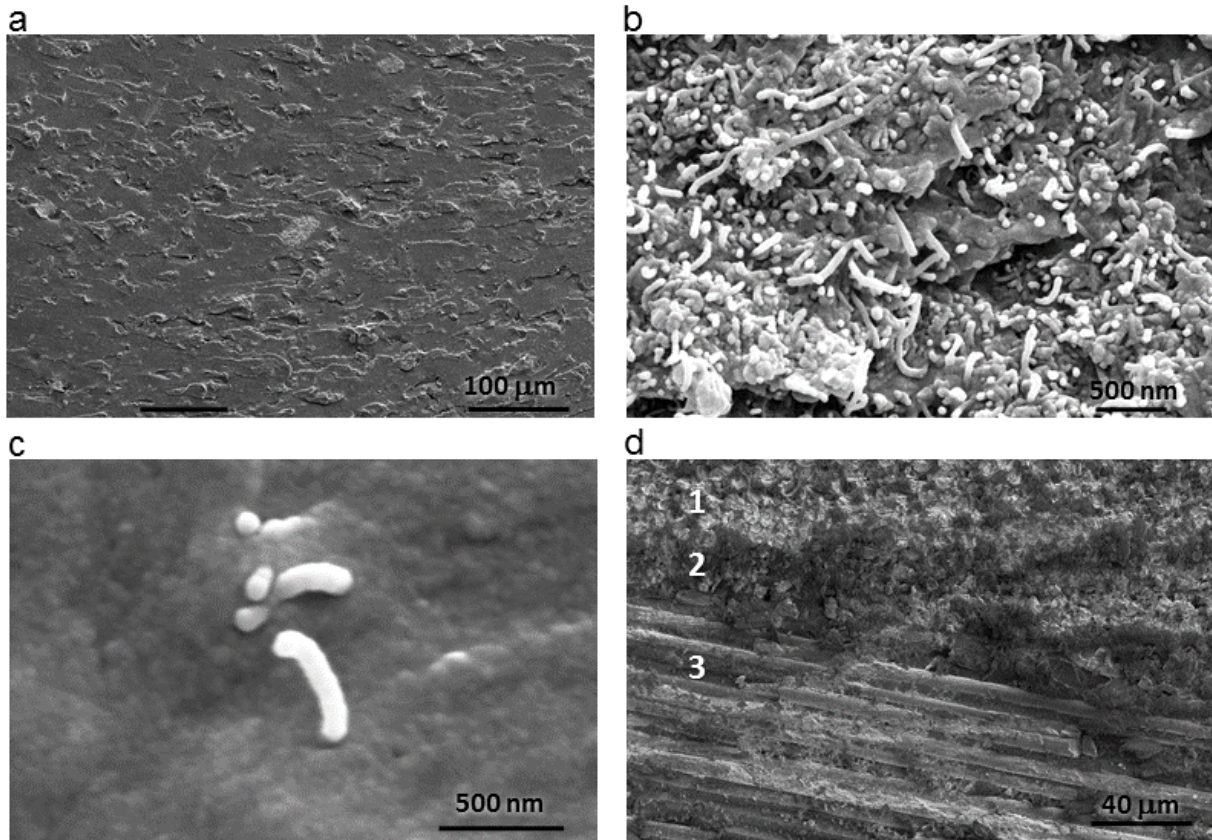


Figure 2. Fracture surface of the Zyvex nanotube matrix material a) agglomerates of CNT are seen as lighter areas b) a high magnification image of the agglomerates c) individual nanotubes protruding from surface d) hybrid composite with Zyvex CNT matrix, carbon fiber (regions 1 and 2) and the N12 nanotube interlaminar layer (region 2).

3.2 Thermal Conductivity

The thermal conductivity results for the first scenario are seen in Table 1. Even a small amount of carbon nanofibers (0.5 wt%) improved the thermal conductivity by 27%. The addition of 1 wt% improved the performance by 59% and the addition of 10 wt% CNF increased thermal conductivity by 400%. The nanofibers are shown to be effective in increasing the thermal conductivity of a polymer, even at low weight loadings. A comparison of 1 wt% CNF and 1 wt% GPL shows similar increases in thermal conductivity, with the GPL composite showing a slightly better increase in thermal conductivity. Figure 3 gives a graphical representation of the thermal conductivity data in Table 1.

Table 1. Epoxy-Based Samples

Sample Description	Thickness (mm)	Density (g/cm ³)	Specific Heat (J/g-K)	Thermal Diffusivity (mm ² /s)	Thermal Conductivity (W/m-K)	Thermal Conductivity at 80°C (W/m-K)
Pure Epoxy	2.196	1.0923	1.3353	0.115	0.131	0.149
0.5% wt% CNF	1.978	0.995	1.2169	0.126	0.167	0.198
1.0% wt% CNF	1.622	1.139	1.2189	0.137	0.208	0.252
10% wt% CNF	1.631	1.169	1.2730	0.334	0.521	0.592
1.0 wt% GPL	1.731	1.192	1.2729	0.149	0.237	0.313

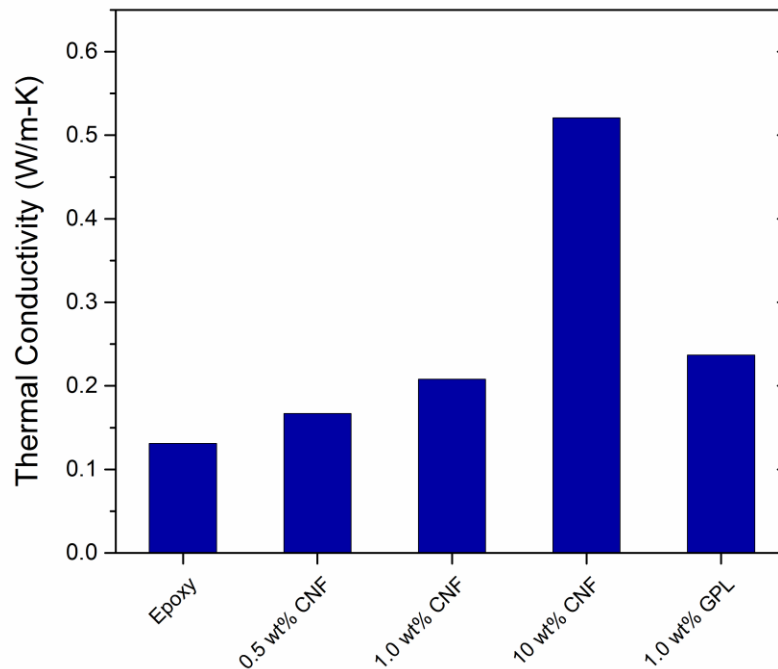


Figure 3. Plot of thermal conductivity for epoxy-based composites with carbon nanofibers (CNF) and graphene platelets (GPL).

The data in Table 2 shows the performance of the hybrid composites, containing both carbon fiber and nanotubes in the structure. First, the neat resin is shown and then the resin with 0.4 wt% CNT. The low volume of nanotubes added results in only a slight increase in the thermal conductivity of the resin. The addition of the continuous carbon fiber greatly increases the thermal conductivity of the composite and the further addition of CNT to the carbon fiber composite, again, only shows a slight increase in performance. As expected, the composite that was cured under pressure in the autoclave had a more dense structure and, consequently, had a higher thermal conductivity. The addition of the interlaminar N12 nanotubes, surprisingly, did not show improved performance over the same composite without them. Because of a test failure, the k of the composite with CNT, CF, N12 and autoclaved had to be estimated based on the improved performance percentage between the oven cured and autoclaved cured Zyvex Resin-CNT-CF material. When the estimated improvement is applied to the Zyvex Resin-CNT-CF-N12 composite, the k is estimated to be 0.902, the highest conductivity of all the materials. This demonstrates that the addition of nanocarbon species can improve performance of a composite, but that the improvement is strongly dependent on the volume fraction of carbon nanomaterial added and the processing methods. The graphical representation of this data is shown in Figure 4.

Table 2. Zyvex Resin-Based Samples

Sample Description	Thickness (mm)	Density (g/cm ³)	Specific Heat (J/g-K)	Thermal Diffusivity (mm ² /s)	Thermal Conductivity (W/m-K)
Zyvex Resin	1.93	1.11	1.59	0.138	0.244
Zyvex Resin-CNT	2.00	1.13	1.58	0.142	0.255
Zyvex Resin-CF	1.28	1.34	1.03	0.494	0.683
Zyvex Resin-CNT-CF	1.15	1.45	1.03	0.467	0.698
Zyvex Resin-CNT-CF autoclave	1.10	1.46	0.981	0.578	0.829
Zyvex Resin-CF-N12	1.20	1.44	0.987	0.567	0.808
Zyvex Resin-CNT-CF-N12	1.17	1.46	1.02	0.510	0.760
Zyvex Resin-CNT-CF-N12-autoclave (est)	-	-	-	-	0.902

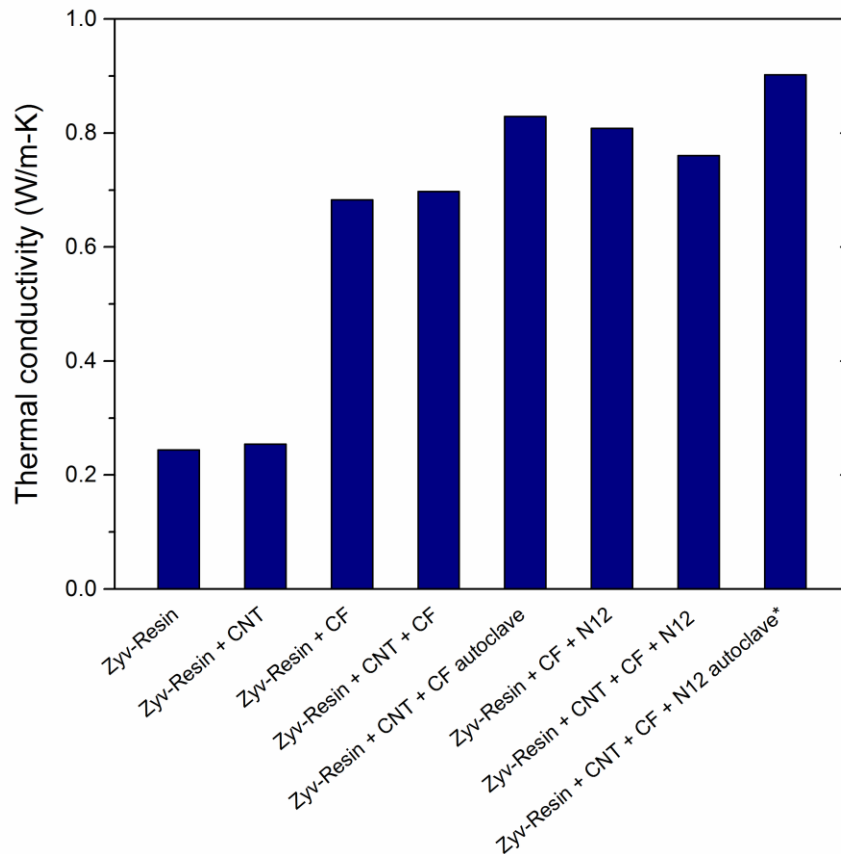


Figure 4. Thermal conductivities of various composite materials based on the Zyvex resin and fiber system, as well as the addition of N12 nanostitch interlaminar carbon nanotubes.

3.3 Thermal Modeling

ABAQUS modeling results are shown in Figure 5. In this model, a thin-walled metallic tube is overwrapped with a PMC consisting of a thermoplastic and carbon fiber, where the composite overwrap is approximately three times thicker than the metallic thin-walled tube. Two thermal models were run, where a typical thermal conductivity for the composite is used in the first run and then a thermal conductivity that is 100% higher is used in the second run. The temperature of the inner diameter (ID), the temperature of the interface between the thin-walled metal tube and the composite, the temperature of the outer diameter (OD) of the composite is calculated after the ID is cyclically exposed to cyclic high temperatures. Compared to Fig. 5a, Fig. 5b shows two interesting trends. First, due to the higher thermal conductivity of the PMC overwrap, the OD reaches a higher temperature at a higher rate than the model in Fig. 5a. This is due to heat conducting more rapidly through the composite. Second, the overall temperature of the ID and the interface is lower for the case with the higher PMC thermal conductivity. The heat from the ID is dissipated more evenly and more quickly throughout the composite structure, helping keep the temperatures lower in these locations. This is important in tubular structures repeatedly

exposed to high temperature gases, a scenario often encountered in armament environments. The temperature is reduced at the interface by approximately 100 °C when the higher thermal conductivity PMC is used, a significant decrease (similar decrease for the max ID temperature as well). The heating cycles per minute decreases after the first two minutes, resulting in the temperature increasing more slowly in both cases. It should be noted that in this generic model, the temperature reached in the composite would be too high an operating temperature for any PMCs, however this model is used to simply make an illustrative point on how composite thermal conductivity could affect a generic structure relevant to armament research.

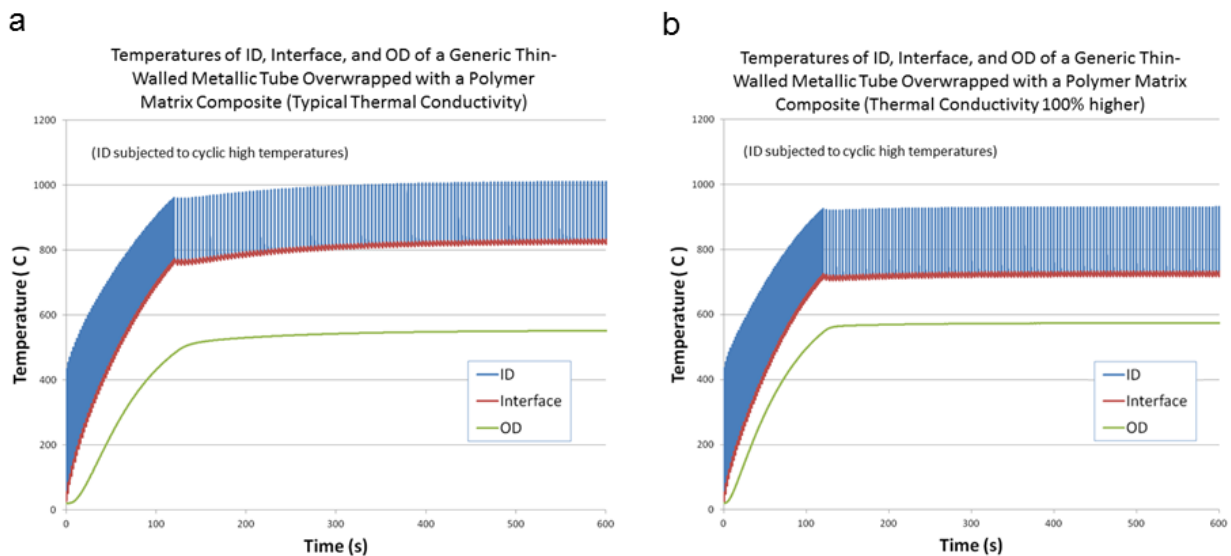


Figure 5. Thermal modeling results for a generic thin-walled metallic tube overwrapped with polymer matrix composite. The ID of the tube is subjected to cyclic heating. The thermal conductivity of the PMC in (b) is 100% higher than the one used in (a).

4. Conclusions

It has been demonstrated that the addition of carbon nanomaterials to polymer matrices can, in some cases, significantly increase the thermal conductivity of the composite. The effectiveness of the carbon nanomaterial additive is dependent upon the particle morphology, weight fraction, and processing conditions. In the case of the hybrid composites, small increases in the thermal conductivity were recorded with the addition of 0.4 wt% nanotubes in the matrix, however, the addition of 10 wt% carbon nanofiber in epoxy resulted in a 400% increase in thermal conductivity. Thermal models showed that increasing the thermal conductivity of the composite in a composite overwrapped thin-walled tube structure by 100% can decrease the ID and interface temperatures by approximately 100 °C, a significant decrease. These tests demonstrate that the addition of carbon nanomaterials to armament composites can make polymer matrix composites a more viable option for armament applications.

5. Acknowledgements

The author acknowledges Dr. Andrew Littlefield for his assistance in the preparation of the hybrid nanotube/carbon fiber composites. The author also acknowledges Mr. Mark Witherell for running the ABAQUS thermal model.

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